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**ACUTE INHALATION TOXICITY
OF CHEMICALLY NEUTRALIZED/HYDROLYZED VX IN RATS**

**SCWO EFFLUENT PRIOR TO EVAPORATION
SCWO EFFLUENT POST-EVAPORATION**

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13. ABSTRACT (Maximum 200 words) The chemical agent VX was neutralized through a series of chemical and oxidative reactions to produce two markedly less toxic effluent streams. The first stream, which came directly from a Supercritical Water Oxidation (SCWO) reaction was identified as SCWO Effluent Prior to Evaporation. The second stream, which went through an evaporation/distillation process after the SCWO reaction was identified as SCWO Effluent Post-Evaporation. An acute inhalation toxicity test was conducted on each of the two effluents per Department of Transportation (DOT) guidelines. Separate groups of Sprague-Dawley rats were exposed for 1 hr to an aerosol concentration of 1.9 mg/L (pre-evaporation effluent) and to a vapor concentration of 5.4 mg/L (post-evaporation effluent). The rats did not exhibit any signs of irritation or overt toxicity during the exposure or post-exposure period. No deaths occurred within the 14-day post-exposure period, indicating that the two effluents were less toxic than a DOT Class 6.1 poison via inhalation. The effluent streams from the SCWO reaction of VX do not appear to pose an acute inhalation hazard.			
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PREFACE

The work described in this report was authorized under the Alternative Technology Program. This work was started in September 1997 and completed in February 1998. The experimental data are recorded in laboratory notebook 97-0093. The storage location for all the raw data and final report are in the Toxicology Archives, Building E-3150.

In conducting the research described in this report, the investigators adhered to the "Guide for the Care and Use of Laboratory Animals," National Institute of Health Publication No. 85-23, 1996, as promulgated by the Committee on Revision of the Guide for Laboratory Animal Facilities and Care of the Institute of Laboratory Animal Resources, National Research Council, Washington, DC. These investigations were also performed in accordance with the requirements of AR 70-18 (Animal Welfare Act), "Laboratory Animals, Procurement, Transportation, Use, Care, and Public Affairs." Final approval for this work was granted by the U.S. Army Edgewood Research, Development and Engineering Center (ERDEC)* Laboratory Animal Use and Review Committee (LAURC) on 6 March 1997 and assigned Protocol No. 98-321.

The performance of this study was consistent with the objectives and standards in "Good Laboratory Practices for Nonclinical Laboratory Studies" (21 CFR 58, Food and Drug Administration, U.S. Department of Health and Human Services, April, 1988).

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* Now known as the U.S. Army Edgewood Chemical and Biological Center (ECBC).

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QUALITY ASSURANCE

This study, conducted as described in the report titled "Acute Inhalation of Chemically Neutralized/Hydrolyzed VX in Rats (SCWO Effluent Prior to Evaporation & SCWO Effluent Post-Evaporation), was examined for compliance with Good Laboratory Practices as published by the U. S. Environmental Protection Agency in 40 CFR Part 792 (effective 17 Aug 1989). The dates of all inspections and the dates the results of those inspections were reported to the Study Director and management were as follows:

<u>Phase Inspected</u>	<u>Date</u>	<u>Date Reported</u>
Data and Final Report	26 Jun 01	26 Jun 01

To the best of my knowledge, the methods described were the methods followed during the study. The report was determined to be an accurate reflection of the raw data obtained.


DENNIS W. JOHNSON 26 Jun 01
Quality Assurance Coordinator
Research and Technology Directorate

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1. INTRODUCTION

VX [O-Ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate, CAS #50782-69-9], is a lethal nerve agent similar in mechanism of action and effects to GB (CAS #107-44-8).¹ Although VX has a low volatility (10.5 mg/M³ @ 25 °C), it is estimated to be twice as toxic as GB by the inhalation route.¹ Due to this toxicity, VX and other agents were produced during the cold war period for possible use as either a chemical warfare agent or as a chemical deterrent. Therefore, chemical weapons storage has resulted in accumulated stockpiles of VX in the United States and other foreign countries.

Public Law 99-145 and subsequent amendments have directed the U.S. Army to dispose of its stockpile of unitary chemical weapons, including VX.² Although incineration was originally the method of choice for disposal, public concern over possible human health risks from air emissions prompted the exploration of alternate technologies. The Alternative Technology Program (ATP) was established to review and develop a process other than incineration to safely dispose of VX.^{2,3}

The method selected by the ATP involves destroying liquid VX through a chemical neutralization (hydrolysis) and oxidative process (Figure 1).⁴ First, VX is chemically neutralized with sodium hydroxide (NaOH, CAS #1310-73-2) to produce a hydrolysate that is high in organics. Second, a Supercritical Water Oxidation (SCWO) unit breaks down the organics in the hydrolysate to form inorganics, primarily salts. Third, the inorganics are removed from the liquid effluent by evaporation/crystallization. This final step leaves behind a collection of salts for solid waste disposal and a clear liquid effluent (distillate) for either discharge into a sewage treatment plant or additional processing.

This report summarizes the procedures and results of an acute inhalation study on the final liquid effluents from an SCWO reaction of the VX/NaOH hydrolysate solution. The liquid streams tested (test material) consisted of the SCWO Effluent - (1) just prior to evaporation (pre-evaporation) and (2) post-evaporation (distillate). The inhalation testing procedures used were based on Department of Transportation (DOT) guidelines in accordance with the Code of Federal Regulation (CFR) 49, Part 173.132 (10/1/94 edition).⁵ Information from this study provided toxicity information on VX neutralized wastestreams as well as helped assign the proper DOT packing group should the test material require transportation following treatment.

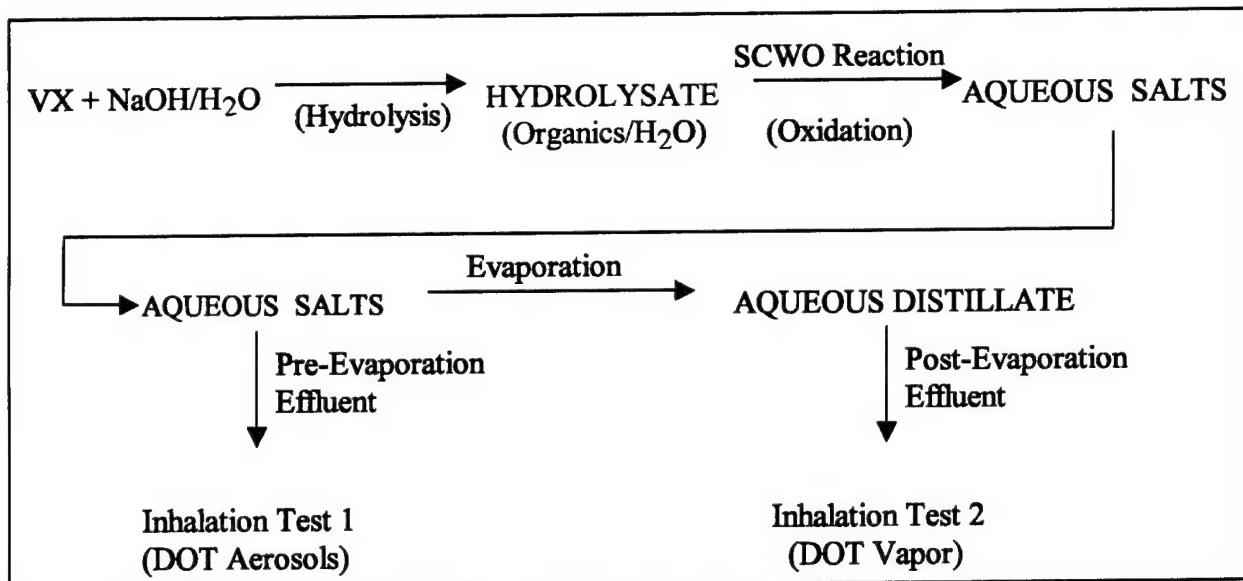


Figure 1. VX Neutralization Process and Inhalation Toxicity on Effluent Streams.

2. MATERIALS AND METHODS

2.1 Test Material.

The U.S. Army Edgewood Research, Development and Engineering Center (ERDEC)* supplied General Atomics Corporation (San Diego, CA) with fifty 1-gal containers of VX/NaOH hydrolysate, which were combined into a single 55-gal drum and fed into an SCWO reactor.⁷ Following the high temperature and pressure reaction, the process fluid was fed into an effluent drum (Drum #4). The SCWO effluent samples from Drum #4 were returned to ERDEC for analytical and toxicological testing. This test solution was designated “SCWO Effluent (prior to evaporation) Drum #4.”

The Environmental Technology Team, ERDEC, produced the post-evaporator effluent by distilling a portion of the Drum #4 effluent. This represented the final step in the VX treatment process. The 3 L test solution was refrigerated at approximately 4 °C prior to use for inhalation testing.

2.1.1 SCWO Effluent Pre-Evaporation.

The test material used in the first inhalation test was identified as neutralized VX, SCWO prior to evaporation, Drum #4. The 1-gal (glass container) of test material consisted of

*Now known as the U.S. Army Edgewood Chemical Biological Center (ECBC).

approximately 85% clear aqueous solution (pH 5.5) as the top layer and approximately 15% inorganic salts (white) as the bottom layer. The test solution was allowed to settle, and the top layer (supernatant), consisting primarily of water and dissolved salts (Na, 12,000 mg/L), was used to generate the exposure atmosphere.

The Analytical Chemistry Team (ACT), ERDEC, found no detectable VX (<20 ppb) in solution using chemical analysis. A summary of the organic and inorganic constituents of this test material is listed in the Appendix.

2.1.2 SCWO Effluent Post-Evaporation

The test material used in the second inhalation test was identified as neutralized VX, SCWO distillate, Drum #4. The test material consisted of a clear aqueous solution (pH 6.6). The sodium concentration was 13 mg/L for the post-evaporation sample analyzed by the Toxicology Team, ERDEC. The total organic carbon on the distillate was <100 ppm.

2.2 Animals

Young adult, male and female Sprague-Dawley rats were obtained from Charles River Laboratories, Incorporated (Wilmington, MA). Animal weight ranges on their respective arrival dates were as follows:

<u>Arrival Date 12/03/97</u> <u>SCWO Effluent Pre-Evaporation</u>	<u>Arrival Date: 1/21/98</u> <u>SCWO Effluent Post-Evaporation</u>
161 - 178 g (5 males)	162 - 175 g (5 males)
177 - 194 g (5 females)	191 - 202 g (5 females)

All animals were quarantined and evaluated for general condition and health status. The animals were identified by permanent marker (tail) and housed in plastic rat cages in the animal holding facility (Building E3222). Housing conditions were 12-hr light/dark cycle with 22 ± 4 °C temperature and 40-70% relative humidity (RH). Certified commercial rodent ration (PMI Feeds, Incorporated, St. Louis, MO) and water were available *ad libitum*, except during testing.

Just prior to testing, all animals were weighed, numbered, and randomly placed into groups. Animal weight ranges on the day of exposure were as follows:

<u>Exposure Date 12/08/97</u> <u>SCWO Effluent Pre-Evaporation</u>	<u>Exposure Date: 1/26/98</u> <u>SCWO Effluent Post-Evaporation</u>
212 - 239 g (5 males)	216 - 226 g (5 males)
196 - 215 g (5 females)	211 - 219 g (5 females)

No controls were required for DOT toxicity testing. However, at the beginning and end of the 14-day study period, one male and one female naive rat were submitted for serological health monitoring for each exposure.

2.3 Inhalation Exposure System (General)

2.3.1 Chamber Operations

All animal exposures were conducted in a 250 L dynamic airflow inhalation chamber. The generation systems (one for aerosols and one for vapor) were placed within a generator box located above the chamber. The SCWO prior to evaporation effluent was generated as an aerosol due to the concentration of dissolved salts. The SCWO post-evaporation effluent was generated as a vapor since this distillate was practically free of aerosol forming organic and/or inorganic constituents. Test atmospheres were generated in the chamber using methods described below. Periodic sampling during the 1-hr exposure was performed to quantify the chamber concentration. Chamber airflow (liters/minute) was measured at the chamber outlet with a thermo-anemometer (Model 8565, Alnor, Skokie, IL). Temperature and RH were monitored using a thermo-hygrometer (Model RH411, Omega, Stamford, CT).

2.3.2 Aerosol Exposure System (SCWO Pre-Evaporation)

The aerosol generation system consisted of a syringe pump, containing the test solution and a spray atomizer (Figure 2). The test solution was drawn up into two 50-mL gas-tight syringes that were mounted onto a variable rate syringe pump (Model 22, Harvard Apparatus, Incorporated, South Natick, MA). The outputs from each syringe were teed onto a plastic line, which extended to a spray atomizer (Spraying Systems Company, Wheaton IL). Once activated, the syringe pump dispensed the test solution at a rate of 1.1 mL/min into the atomizer situated at the chamber inlet. Compressed air (30 psig) directed through the side of the atomizer forced the test solution into fine aerosol droplets, which were drawn down into the chamber. The combination syringe drive dispense rate and chamber flow gave a nominal chamber concentration of 2.0 mg/L.

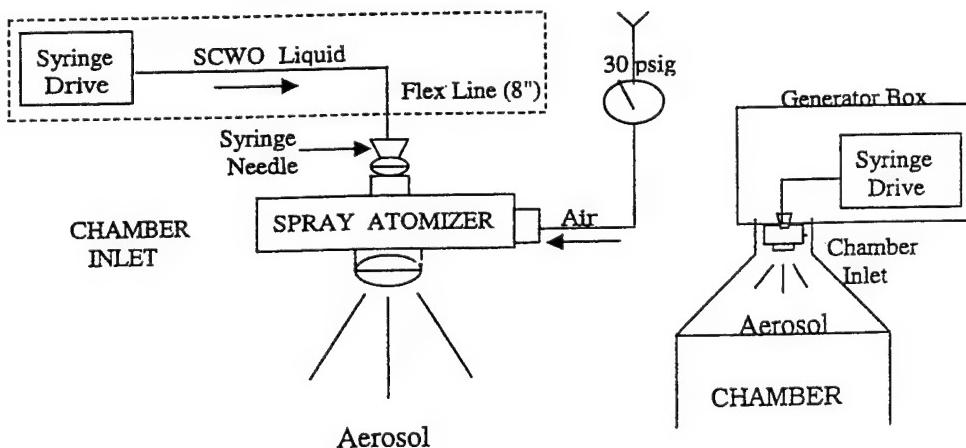


Figure 2. Aerosol Generation System and Inhalation Chamber.

2.3.3 Vapor Exposure System (SCWO Post-Evaporation).

The vaporization system for the SCWO post-evaporator effluent consisted of a reservoir, liquid flow pump, and heated manifold to deliver and vaporize the solution into the chamber (Figure 3). The test solution was placed into a glass reservoir located in the generator box above the chamber. A fluid metering pump (Model QSYX-QO-SSY, Fluid Metering Incorporated, Oyster Bay, NY) delivered a set flow (1.73 mL/min) of test solution from the reservoir into a heated manifold (98 °C). The manifold consisted of a ¼-in. cross Swagelok® fitted with a 50 W heater element. Vapor from the manifold entered a glass mixing bowl situated at the chamber inlet. Upon activation of the system, a combination of metered liquid, heating and purge air (3.5 L/min) sent vaporized effluent to the chamber.

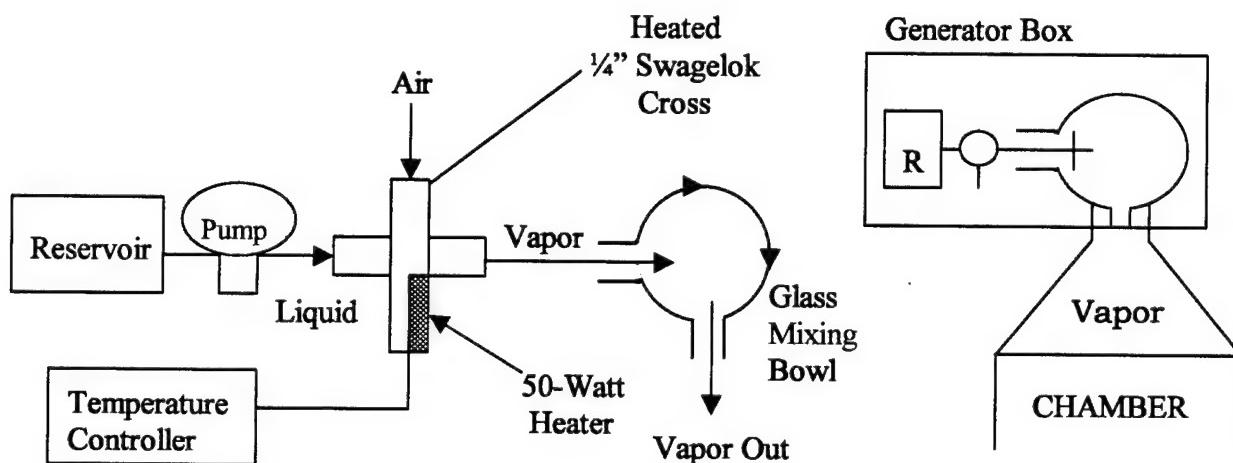


Figure 3. Vapor Generation System and Inhalation Chamber.

2.4 Acute Inhalation Animal Exposures.

2.4.1 SCWO Pre-Evaporation.

Five male and five female Sprague-Dawley rats were placed in stainless steel compartmentalized cages (20 in. x 14 in. x 4 in.) within the chamber. The rats were exposed whole body to an aerosol concentration of 2 mg/L for 1 hr per DOT guidelines (Table 1). All animals were observed for toxic signs resulting from the exposure. Following exposure, the rats were returned to their holding room for observation during the 14-day post-exposure period.

Table 1. DOT Hazard Classification and Packaging Categories for Division 6.1 Mixtures*

Inhalation Toxicity Testing	
Packaging Group	Inhalation Toxicity by Dusts and Mists LC ₅₀ (mg/L)
I	≤ 0.5
II	> 0.5, ≤ 2
III	> 2, ≤ 10

*The mixture is classified as a 6.1 inhalation poison if lethality occurs in half of the animals tested within 14 days post-exposure.⁵

2.4.2 SCWO Post-Evaporation

Five male and five female Sprague-Dawley rats were exposed whole body to a vapor concentration of 5 mg/L (6,800 ppm) for 1 hr. This vapor concentration targeted the DOT packing group III category (least toxic) for a vapor exposure. During the exposure period, the animals were observed for toxic signs resulting from inhalation of the test material. Following exposure, the rats were returned to their holding room for observation for the 14-day post-exposure period.

2.5 Sample Collection and Analysis

2.5.1 SCWO Pre-Evaporation

Aerosol concentrations of the SCWO effluent in the chamber were determined by collecting filter pad samples (PTFE, 25 mm Gelman, Ann Arbor, MI) during exposure. The filter pads were mounted onto open-faced filter holders (25 mm) connected to a vacuum. Sample flows (2 L/min) were measured using a constant flow air sampler with rotameter (Sierra, Carmel Valley, CA). Samples were drawn from the chamber at set intervals (5, 15, 25, 35, 45, and 55 min) during exposure. Sample filters were then desorbed with 18 MΩ of deionized water and subsequently analyzed for sodium by flame atomic absorption spectroscopy. The measured aerosol concentration in the chamber atmosphere was based on the collection and analysis of sodium, a major component in the SCWO effluent. Computations on the amount of sodium in the chamber were then used to determine the total aerosol concentration. A linear regression fit ($R^2 \geq 0.999$) of diluted sodium standards (absorbance versus concentration) was used to compute for sample quantitation.

The aerodynamic particle size was measured using a 10-stage cascade impactor (model 2210-K, Graseby-Andersen, Atlanta, GA). Impactor samples were drawn for 8 min (7 L/min) during the midpoint of the exposure. Aerosols drawn through the impactor were collected onto glass fiber substrates beneath each stage. The substrates were placed into a

dessicator (2 hr) prior to and after sampling to avoid moisture weight gain and to determine the mass of dried material (primarily sodium) on each substrate. Each substrate was subsequently weighed to determine mass collected at each size range. Particle size sample data was analyzed by log-normal regression (least squares method) of particle size versus cumulative relative mass to determine mass median aerodynamic diameter (MMAD) and geometric standard deviation (σ_g).

2.5.2 SCWO Post-Evaporation.

Vapor concentrations of the SCWO effluent in the chamber were determined by measuring the temperature and % RH every 1-2 min during exposure. Temperature and % RH measurements were conducted using a calibrated Thermo Hygrometer (Model RH 411, Omega, Stamford, CT). These measurements were used to convert to the vapor pressure of water from standard tables listed in the CRC Handbook.⁶ The amount of water vapor (milligrams per liter) present in the chamber was computed using the following gas constant formula:

$$PV = nRT$$

$$\text{or } PV = g/(MW) RT$$

$$\text{or } g/V = P \times MW/RT$$

$$\text{or } \text{mg/L (H}_2\text{O)} = \frac{(P)(MW)}{(R)(T)} \times 1000 \text{ mg/g}$$

where P = (torr or mm Hg) water vapor pressure determined from standard table

MW = 18.02 g/mole H_2O

R = 62.4 $\frac{(mm\ Hg)\ (L)}{(mole)\ (^{\circ}K)}$

T = $^{\circ}K$

Temperature and % RH measurements were taken before and after exposure to determine the amount of background water vapor in the chamber. Background measurements were taken with the rats in the chamber to account for animal respiration and body heat. The amount of water vapor in the chamber due to the test solution was determined by subtracting the total vapor in the chamber during exposure from the background water vapor.

For the post-evaporative solution, filter pad samples were also collected during the chamber calibration runs to ensure that vapors and not aerosols were present in the chamber atmosphere.

3. RESULTS

3.1 Aerosol Concentration During Exposure (SCWO Pre-Evaporation).

The mean sodium concentration during the 1 hr exposure was $22.4 \mu\text{g/L} \pm 1.5$ (Table 2). This equated to a total aerosol concentration of $1.9 \text{ mg/L} \pm 0.1$ based on calculations of nominal chamber concentrations (for aerosol and sodium) versus the analytical sodium concentration (Table 3). The chamber flow during exposure was 511 L/min with a chamber equilibration time (t_{99}) of 2.3 min.

3.2 Aerosol Particle Size.

The MMAD was $3.26 \mu\text{m}$, and the geometric standard deviation (σ_g) was 2.60, indicating a polydispersed aerosol (Table 2). More than 90% of the particles were within the respirable range ($<10 \mu\text{m}$) for particle deposition in the lung.

Table 2. Sodium Concentration and Particle Size During 1-Hour Exposure to Neutralized VX (SCWO Pre-Evaporation).

	Sample Time (min)	Sodium (Na) ($\mu\text{g/L}$)	Particle Size	
			MMAD (μ)	σ_g
Filter Pads	5 - 8	25.1	3.26	2.60
	15 - 18	21.8		
	25 - 28	20.8		
Cascade Impactor	25 - 33			
Filter Pads	35 - 38	21.8	3.26	2.60
	45 - 48	22.5		
	55 - 58	22.1		
Mean Na = $22.4 \mu\text{g/L} \pm 1.5$; CV = 6.5%				

Table 3. Total Aerosol Chamber Concentration for the SCWO Pre-Evaporation Exposure

Sodium in Chamber ($\mu\text{g/L}$) (Nominal)	$= \frac{12,000 \text{ mg/L (Na in test solution)} \times (0.0011 \text{ L/min}) \times 10^3 \text{ g/mg}}{511 \text{ L/min (Chamber Flow)}}$ $= 25.8 \mu\text{g/L Nominal Na}$
Total Aerosol Concentration (mg/L) (Nominal)	$= \frac{1.1 \text{ mL/min (dispense rate)} \times 1.0 \text{ g/mL (density)} \times 10^3 \text{ mg/g}}{511 \text{ L/min (Chamber Flow)}}$ $= 2.2 \text{ mg/L Nominal Aerosol}$
Total Aerosol Concentration (Based on Sodium Level)	$\frac{25.8 \mu\text{g/L Na (nominal)}}{2.2 \text{ mg/L (total aerosol nominal)}} = \frac{22.4 \mu\text{g/L Na (analytical)}}{x \text{ mg/L (total aerosol)}}$ $= 1.9 \text{ mg/L Total Aerosol}$

3.3 Water Vapor Concentration During Exposure (SCWO Post-Evaporation).

The water vapor concentration during the 1-hr exposure was 5.4 mg/L SCWO effluent. Chamber flow was 330 L/min with a chamber equilibration time (t_{90}) of 3.5 min. The nominal water vapor concentration from the chamber operating parameters was 5.2 mg/L. This corresponded to a 102% recovery of water vapor in the chamber. Calculations for water vapor concentrations and percent recovery are listed in Table 4.

3.4 Toxicology.

3.4.1 SCWO Pre-Evaporation.

Animals were monitored for toxic signs and behavioral changes during exposure to the aerosols from the SCWO prior to evaporation effluent. Animals showed no toxic signs during either the exposure or post-exposure period. All animals showed a normal increase in weight, and there were 0/10 deaths during the 14-day post-exposure period. Since no mortality occurred at the upper exposure level (Packaging Group III), no further testing was required.

3.4.2 SCWO Post-Evaporation.

Animals were monitored for toxic signs and behavioral changes during exposure to the SCWO distillate. Animals showed no toxic signs during either the exposure or post-exposure period. All animals showed a normal increase in weight, and there were 0/10 deaths during the 14-day post-exposure period. Since no mortality occurred at the upper exposure level (Packing Group III), no further testing was required.

Table 4. Water Vapor Concentration in the Exposure Chamber to SCWO Effluent Post-Evaporation and Percent Recovery

Total H ₂ O Vapor in Chamber (mg/L) (During Exposure)	= $\frac{(10.751 \text{ mm Hg}) \times (18 \text{ g/mole H}_2\text{O}) \times 10^3 \text{ mg/g}}{62.4 \text{ mm Hg (L)} \times 293.9 (\text{°K})}$ = 10.6 mg/L
Background H ₂ O Vapor in Chamber Measured Before and After Exposure (mg/L)	= $\frac{(5.277 \text{ mm Hg}) \times (18 \text{ g/mole H}_2\text{O}) \times 10^3 \text{ mg/g}}{62.4 \text{ mm Hg (L)} \times 293.9 (\text{°K})}$ = 5.2 mg/L
H ₂ O Vapor in Chamber (mg/L) Due to SCWO Effluent	= 10.6 (total) - 5.2 (background) = 5.4 mg/L
H ₂ O Vapor Concentration (mg/L) (Nominal)	= $\frac{1.73 \text{ mL/min} \times 1.0 \text{ g/mL (density)} \times 10^3 \text{ mg/g}}{330 \text{ L/min (Chamber Flow)}}$ = 5.2 mg/L
% Recovery	$\frac{(5.4 \text{ mg/L H}_2\text{O Vapor in the Chamber}}{(5.2 \text{ mg/L H}_2\text{O Vapor (nominal)}} \times 100 = 102\%$

4. DISCUSSION

Two effluents from a VX neutralization (SCWO) reaction were tested for acute inhalation toxicity per DOT guidelines.⁶ The guidelines specify exposing groups of rats to different concentration levels to establish a relative toxicity level and corresponding packing group for transportation. In this study, only the highest concentration levels (Category III - least toxic) required testing to establish the DOT packing category.

The first inhalation exposure involved testing the SCWO effluent pre-evaporation. The test solution contained a high dissolved sodium concentration (either 12,000 ppm or 1.2%), which was easily generated as an aerosol, however, limited the maximum exposure concentration to 2.0 mg/m³. Higher aerosol concentrations either clogged the spray atomizer (generator) or produced larger particles outside the respirable range. During the exposure, the amount of

sodium in the aerosol generator may have caused the exposure concentration to drop slightly to 1.9 mg/L instead of attaining 2.0 mg/L. However, this slight decrease should not have affected the experimental outcome since the animals did not exhibit any signs of either irritation or toxicity during exposure.

The second inhalation exposure involved testing the SCWO effluent post-evaporation. Due to the lack of significant inorganic and organic constituents in the SCWO effluent (distillate), this particular solution did not lend itself to form an aerosol as did the pre-evaporation solution. A vapor exposure was used because it was easier to generate a higher vapor concentration as opposed to an aerosol, which in turn would allow for a better assessment of the inhalation toxicity of the material. Although the measured water vapor in the chamber was based on calculations from temperature and RH readings, the calculated concentration corresponded well with the nominal concentration. The animals showed no toxic signs at the highest distillate concentration obtainable with the configured chamber system (5.1 mg/L or 6,680 ppm H₂O vapor).

The toxicological characterization of the SCWO effluent streams from the VX/NaOH reaction, as assessed via inhalation exposure, showed no animal mortality or overt toxicity at high exposure levels. Based on these findings, the effluent wastestreams (pre- and post-evaporation) were less toxic than a Group III, Class 6 poison.

5. CONCLUSIONS

Based on the findings of this study, the following conclusions can be made:

- The aerosol inhalation toxicity of the reaction product (SCWO effluent prior to evaporation) from neutralized VX was less toxic than "Packing Group III materials" according to biological criteria set forth in Department of Transportation CFR 49 (Part 173.132 - 173.133, Class 6, Division 6.1, pages 504-508, October 1, 1994).
- The vapor inhalation toxicity of the reaction product (SCWO effluent post-evaporation) from neutralized VX was less toxic than "Packing Group III materials" according to biological criteria set forth in Department of Transportation CFR 49 (Part 173.132 - 173.133, Class 6, Division 6.1, pages 504-508, October 1, 1994).
- The SCWO reactor effluents (just prior to and post-evaporation) from the VX neutralization process do not appear to pose an acute inhalation hazard.

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LITERATURE CITED

1. *Chemical Agent Data Sheets*, Vol. 1, EA-SR-74001, pp 167-179, Edgewood Arsenal, MD, December 1974, UNCLASSIFIED Report (AD B028 222).
2. *Detailed Program Plan and Methodology for Developing Alternative Technologies for Chemical Demilitarization*, U.S. Army Chemical Demilitarization and Remediation Activity, Aberdeen Proving Ground, MD, December 5, 1994.
3. *Alternative Technologies for the Destruction of Chemical Agents and Munitions*, National Research Council, Committee on Alternative Chemical Demilitarization Technologies, National Academy Press, Washington, DC, 1993.
4. *Test and Evaluation Master Plan for NECDF Chemical Neutralization Followed by Supercritical Water Oxidation (SCWO)*, Draft Report, U.S. Army Chemical Demilitarization and Remediation Activity, Aberdeen Proving Ground, MD, February 16, 1999.
5. Code of Federal Regulations, 49 CFR (Transportation), Parts 100 - 177, Sections 173.132 - 173.133, Class 6, Division 6.1, pp 504-505, October 1, 1994.
6. *Handbook of Chemistry and Physics (CRC)*, 49th ed., p D-109, The Chemical Rubber Publishing Company, Cleveland, OH, 1968.

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APPENDIX

PRIMARY COMPONENTS OF SCWO EFFLUENT PRE-EVAPORATION (DRUM # 4)

	Solid Phase	Liquid Phase
(Metals) Results in ppm		
Calcium (CAS #7440-70-2)	11,951	4
Iron (CAS # 7439-89-6)	1,248	< 0.07
Magnesium (CAS # 7439-95-4)	301	< 0.03
Phosphorus (CAS # 7723-14-0)	172,664	4,708
Sodium (CAS # 7440-23-5)	173,867	12,215
Titanium (CAS # 7440-32-6)	14,300	0.8
(Volatile Organics)		
VOC Analysis (Headspace)		
Chloroform (CAS # 67-66-3)		< 20.0 µg/L
SVOC Analysis (Purge & Trap)		
Chloroform		300 µg/L
Methylphosphonic Acid (MPA) (CAS # 993-13-5)		6 ppm
Organics (Chloroform Extracted)		
Diethyl Phthalate (CAS # 84-66-2)		170 mg/L
Tributyl Phosphate (CAS # 126-73-8)		< 1% (too low to quantitate)
Dibutyl Phthalate (CAS # 84-74-2)		26 mg/L